भारतीय मानक
Indian Standard

IS 2600 (Part 5) : 2022

डाई कॉस्टिग के लिए जिंक एवं जिंक आधारित मिश्रणों के रासायनिक विश्लेषण की विधियाँ

भाग 5 इन्डक्टिवली कपल्ड आप्टिकल उत्सर्जन स्पेक्ट्रोमेट्री

(पहला पुनरीक्षण)

Methods of Chemical Analysis of Zinc and Zinc Base Alloys for Die Castings

Part 5 Analysis by Inductively Coupled Plasma Optical Emission Spectrometry

(First Revision)

ICS 77.120.60

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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 5) (First Revision) was adopted by Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 2600 was first published in 1964 and has been revised (in parts) on the basis of implementation of the standard by the manufacturers and the testing.

This standard (Part 5) cover the methods for the analysis of chemical composition of zinc and zinc based alloys for die castings by inductively coupled plasma optical emission spectrometry. The other parts of the series are as follows:

Part 1 Determination of copper, iron, nickel, tin and thallium by spectrophotometric
Part 2 Determination of copper, iron, lead and cadmium by atomic absorption spectrometric method
Part 3 Determination of indium by atomic absorption spectrometric method
Part 4 Determination of chromium by spectrometric method
Part 6 Determination of magnesium by atomic absorption spectrometric method
Part 7 Determination of aluminium by titrimetric method

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ZINC AND ZINC BASE ALLOYS FOR DIE CASTINGS

PART 5 ANALYSIS BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY

(First Revision)

1 SCOPE

This standard (Part 5) covers inductively coupled optical emission spectrometric method for the analysis of chemical composition of zinc and zinc alloys in the range as specified in IS 209, IS 713 and IS 742.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subjected to revisions and parties to agreement based on these standards are encouraged to investigate the possibility of applying the most recent edition of the standards indicated below:

IS No.	*Title*
209 : 1992	Zinc ingot — Specification (*fourth revision*)
265 : 2021	Hydrochloric acid (*fifth revision*)
713 : 1981	Zinc based alloy ingot for die casting (*second revision*)
742 : 1981	Zinc based alloy die casting (*second revision*)
1817 : 1961	Methods of sampling non-ferrous metals for chemical analysis

3 SAMPLING

The samples shall be drawn and prepared in accordance with the procedure laid down in IS 1817.

4 QUALITY OF REAGENTS

4.1 Unless otherwise specified, analytical grade reagents and distilled or demineralized water shall be employed in the test.

4.2 Reagents

4.2.1 *Hydrochloric Acid*, rd (Relative Density) = 1.16 (*see* IS 265).

4.2.2 *Nitric Acid*, rd = 1.4.

4.2.3 *Tartaric Acid*, solid.

4.2.4 *Tartaric Acid Solution*, 25 g/l.

4.2.5 *Zinc*, purity 99.999 percent, free of lead, cadmium, iron, tin copper, magnesium, nickel, chromium, thallium and indium.

4.2.6 *Aluminium Nitrate Monohydrate*, heavy metal impurities: lead, 0.002 percent *Max*, iron, 0.001 percent *Max*.

4.2.7 *Acid Mixture*

Dissolve 1 g of tartaric acid in about 500 ml of water in a 1 000 ml volumetric flask. Add 200 ml of nitric acid, dilute to the mark with water and mix.

4.2.8 *Zinc Solution*

Prepare a solution of 100 g/l by carefully dissolving 100 g of zinc, weighed to the nearest 0.1 g, with a minimum of nitric acid added in small increments. Transfer to a 1 000 ml volumetric flask, add 40 ml of tartaric acid solution (*see* **4.2.4**), cool to room temperature, dilute to the mark with water and mix. It is recommended to verify the purity of the zinc used, to check the presence of the analyze elements present. If any analyze element is present, determine its concentration by the standard addition method and take this concentration into account in the calibration standards.

4.2.9 *Mono-element Standard Solution*

Prepare solutions of 1 g/l of lead, cadmium, iron, copper, aluminium and magnesium, by dissolving

1 g of each of these metals, weighed to the nearest 0.001 g, in the acid mixture. (*see* **4.2.7**) and making up to volume with the acid mixture. (*see* **4.2.7**). Prepare a solution of 1 g/l of tin, by carefully adding 100 ml of hydrochloric acid to 1 g of pure tin. When dissolution is complete, cool and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix. By dilution, prepare standard solutions of 100 mg/l and 10 mg/l, if necessary.

4.2.10 *Aluminum Standard Solution*

Prepare a solution of 50 g/l by dissolving 695.2 g of aluminium nitrate nonahydrate (*see* **4.2.6**), weighed to the nearest 0.1 g, in water. Transfer to a 1 liter volumetric flask, add 40 ml of tartaric acid solution (*see* **4.2.4**), dilute to the mark with water and mix.

4.2.11 *Interenal Standard Solution*

Weigh 6.25 g of Y_2O_3 (yttrium oxide p.a.), to the nearest 0.01 g, and transfer into a 5 litre volumetric flask. Add about 2.5 litre of water, 250 ml of nitric acid and warm gently until dissolution. Cool to room temperature, dilute to the mark with water, and mix.

5 METHOD OF ANALYSIS

A sample of the metal or alloy is dissolved in a mixture of nitric and tartaric acid, and after adequate dilution and atomization of the solution in inductively coupled plasma. Plasma is a high-temperature emission source generated by an radiofrequency (RF) field consisting of a mixture of argon atoms, argon ions and electrons. Sample atoms entering the plasma emit light radiation, whose characteristic wavelengths and intensities are used to identify the elements and determine concentrations, respectively.

6 PROCEDURE

6.1 Preparation of Sample Solution

Weigh 10 g of the sample to the nearest 0.001 g. Transfer the sample into a 500 ml beaker and cover with a watch glass. Dissolve the sample by adding 10 ml of tartaric acid solution (*see* **4.2.4**) and 50 ml of nitric (*see* **4.2.2**). After dissolution, transfer quantitatively to a 250 ml standard flask, add 10 ml of internal standard solution (*see* **4.2.11**), cool to room temperature and made up to the mark with water and mix.

Table 1 Composition of Spectrometer Calibration Solution

(*Clauses* 6.2 *and* 6.2.1)

Sl No.	Calibration Standard No.	1	2	3	4	5	6	7
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
i)	Alloying element 1	0.8 m	M	(3 m + M)/4	(m + M)/2	(m + 3M)/4	M	1.2 M
ii)	Alloying element 2	1.2 M	M	(m + 3M)/4	(m + M)/2	(3m + M)/4	M	0.8 m
iii)	Alloying element 3	(3 m + M)/4	M	0.8 m	(m + M)/2	1.2 M	M	(m + 3M)/4
iv)	Alloying element 4	(m + 3M)/4	M	1.2 M	(m + M)/2	0.8 m	m	(3m + M)/4
v)	Impurity element 1	Blank	0.2 L	0.5 L	0.8 L	L	1.2 L	1.5 L
vi)	Impurity element 2	0.2 L	0.5 L	0.8 L	L	1.2 L	1.5 L	Blank
vii)	Impurity element 3	0.5 L	0.8 L	L	1.2 L	1.5 L	Blank	0.2 L
viii)	Impurity element 4	0.8 L	L	1.2 L	1.5 L	Blank	0.2 L	0.5 L
ix)	Impurity element 5	L	1.2 L	1.5 L	Blank	0.2 L	0.5 L	0.8 L
x)	Impurity element 6	1.2 L	1.5 L	Blank	0.2 L	0.5 L	0.8 L	L
xi)	Impurity element 7	1.5 L	Blank	0.2 L	0.5 L	0.8 L	L	1.2 L
xii)	Remainder Zn	(100 – sum)	(100 – sum)	(100 – sum)	(100 – sum)	(100 – sum)	(100 – sum)	(100 – sum)

where

m = minimum content specification for alloying element percent (mass fraction).

M = maximum content specification for alloying element percent (mass fraction).

L = maximum content specification for impurity element percent (mas fraction).

NOTES

Rounding rules:

1 For contents > 1 percent (mass fraction), 2 numbers behind the comma, the last number being 5 or 0.

2 For contents < 1 percent (mass fraction), round to 2 significant numbers, the last Number being 5 or 0.

3 Do not apply rounding rules for contents < 0,001 5 percent (mass fraction).

6.2 Calibration

Prepare the calibration solutions taking into account the product specification on chemical composition, in accordance with Table 1.

6.2.1 Into each of seven 250 ml volumetric flasks, transfer appropriate volumes (between 1 ml and 50 ml) of the relevant solutions (*see* **4.2.8, 4.2.9** and **4.2.10**). Calculate each volume to contain the exact mass of analyze, such as to give a total mass of 10 g, in order to obtain direct reading in percent (mass fraction) in accordance with Table 1. Add 10 ml of internal standard solution (*see* **4.2.11**), dilute to the mark with acid mixture (*see* **4.2.7**), and mix.

6.2.2 Use internal standardization and background correction techniques, if these techniques optimize the calibration graphs. The correlation coefficient of each calibration graph should be at least 0.999.

In order to validate the calibration graphs, it is recommended to analyze a certified reference material (CRM), if available

6.3 Analysis

Carry out the analysis by optical emission spectrometry with plasma source, using the sample solution prepared as per **6.1**. For the analysis of the impurities and alloying elements in zinc and zinc alloys, as defined in IS 209, IS 713 and IS 742, by optical emission spectrometry, different analytical lines can be used. A list of the wavelengths of appropriate analytical lines is given in Table 2.

The selection of analytical lines used depends on the analytical program and the type of spectrometer.

Perform the analysis at least two times. If malfunction of the spectrometer is suspected, perform additional tests.

A list of analytical lines commonly used for the analysis of zinc and zinc alloys by inductively coupled plasma optical emission spectrometry has been provided in Table 2.

Table 2 List of Analytical Lines

(*Clause* 6.3)

Sl No.	**Element**	**Analytical Lines** Nm
(1)	(2)	(3)
i)	Pb	283.306; 220.353
ii)	Cd	226.502
iii)	Fe	259.940
iv)	Cu	324.754
v)	Sn	189.989; 303.412
vi)	Al	257.510
vii)	Mg	279.553
viii)	Y	321.669

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website- www.bis.gov.in or www.standardsbis.in.

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